

Claims

What is claimed is:

1. A method of making crystals, comprising:
 - suspending a porous preform made of a crystal raw material in a hermetically-sealed chamber;
 - heating the porous preform to a selected treatment temperature that enables reaction between a fluorinating agent and oxide impurities in the porous preform;
 - reacting the fluorinating agent with the oxide impurities at the selected treatment temperature for a selected time period;
 - forming a melt from at least a portion of the porous preform;
 - filling a crucible in the hermetically-sealed chamber with the melt; and
 - progressively moving the melt through a temperature gradient zone defined inside the hermetically-sealed chamber to grow one or more crystals.
2. The method of claim 1, wherein the hermetically-sealed chamber is provided by a hermetically-sealed muffle furnace made of a non-porous refractory material.
3. The method of claim 2, wherein the non-porous refractory material comprises one selected from the group consisting of quartz, alumina, silicon carbide, vitreous carbon, vitreous graphite, glass carbon-coated graphite, and combinations thereof.
4. The method of claim 1, wherein the fluorinating agent is in gaseous form and is selected from the group consisting of CF_4 , NF_3 , SF_6 , BF_3 , C_2H_4 , HF , F_2 , and mixtures thereof, and similar fluorinating gases known in the art.
5. The method of claim 4, wherein the fluorinating agent is supplied into the hermetically-sealed chamber in a stream of inert gas.
6. The method of claim 4, wherein the pressure in the hermetically-sealed chamber while reacting the fluorinating agent with the porous preform is in the range of 0.1 to 5 atm.

7. The method of claim 4, wherein the porous preform is heated to a treatment temperature in the presence of an inert gas prior to exposing the porous preform to the gaseous fluorinating agent.
8. The method of claim 1, wherein reacting the fluorinating agent with the oxide impurities comprises removing volatile gases formed during reaction of the fluorinating agent with the oxide impurities from the hermetically-sealed chamber, and wherein forming the melt occurs in an inert atmosphere.
9. The method of claim 1, wherein the fluorinating agent is in solid form and mixed into the crystal raw material in a range from 0.5 to 2 wt%.
10. The method of claim 1, wherein progressively moving the melt occurs in an inert atmosphere or vacuum.
11. The method of claim 1, wherein the crystal raw material comprises a metal fluoride selected from the group consisting of CaF_2 , BaF_2 , MgF_2 , SrF_2 , LiF , NaF , $(\text{M}_1)_x(\text{M}_2)_{1-x}\text{F}_2$, and M_3AlF_6 , and mixtures thereof; and where M_1 is selected from the group consisting of Li, K, and lanthanide series metal fluorides; M_2 is selected from the group consisting of Ca, Ba, Mg, Sr, and lanthanide series metal fluorides; M_3 is selected from the group consisting of Li, Na, K, Rb, and Cs; and x is in a range from 0 to 1.
12. The method of claim 11, wherein the crystal raw material further comprises a lanthanide series metal fluoride mixed with the metal fluoride.
13. The method of claim 1, wherein the selected time period is in the range of 1 to 240 hours, and the selected treatment temperature is dependent on the melting point of the crystal raw material and is in the range of 50 to 500 °C below the melting point of the crystal raw material.

14. An apparatus for making crystals, comprising:
 - a hermetically-sealed muffle furnace made of a non-porous refractory material;
 - at least one port for entry and exit of gaseous substance within the muffle furnace;
 - at least two temperature-controlled zones defined inside the muffle furnace;
 - a crystal growth crucible disposed inside the muffle furnace;
 - an actuator operable to translate the crucible along a length of the muffle furnace; andmeans for suspending a porous preform above the crucible.
15. The apparatus of claim 14, wherein the non-porous refractory material comprises one selected from the group consisting of quartz, alumina, silicon carbide, vitreous carbon, vitreous graphite, glass carbon-coated graphite, and combinations thereof.
16. The apparatus of claim 14, wherein the crucible is made of a non-porous refractory material selected from the group consisting of quartz, alumina, silicon carbide, vitreous carbon, vitreous graphite, glass carbon-coated graphite, and combinations thereof.
17. The apparatus of claim 14, wherein the at least one port is provided in an end cap fitted in sealed contact at an end of the muffle furnace, and wherein the cap is made of a non-porous refractory material selected from the group consisting of quartz, alumina, silicon carbide, vitreous carbon, vitreous graphite, glass carbon-coated graphite, and combinations thereof.
18. The apparatus of claim 14, wherein the actuator is operable to rotate the crucible about the axial axis of the muffle furnace.
19. The apparatus of claim 14, wherein the crucible comprises one or more crystal growth chambers stacked vertically.
20. The apparatus of claim 14, wherein the temperature-controlled zones are defined by one or more heaters disposed external to the muffle furnace, one or more conductive portions of the muffle furnace, and combinations thereof.